

# Developing Tests to Detect Adulteration of Honey<sup>1</sup>

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First, may I thank your program chairman, Binford Weaver for giving me the privilege of telling you where we stand in our research program. If you get the idea from the title that we have not yet solved all of our problems, you are correct. We have been working toward the principal objective for about 18 months now; the past year has been most challenging, stimulating, and enjoyable.

As I suppose most of you know, the main objective in our research program has been to find ways to detect the illegal addition of high-fructose corn sirup (HFCS) to honey. This fairly new industrial sweetener is very highly refined, contains the same sugars as honey, in slightly different proportions, and is produced in very large amounts and sold at low prices. While I am on the subject of HFCS let me update you on some of the more recent developments in this area. The first great expansion of production seems to be about complete, with six current United States producers, plus one or two more in 1977. Three firms have deferred plans to build HFCS plants, largely because of low sugar prices. The 1976 production was expected to be about 1 million tons dry basis, but was probably closer to 800,000 tons. Whether production will reach the 2,200,000 tons projected for 1977 remains to be seen.

When I spoke to you at Philadelphia last January, I said the price had decreased from 24 to 18 cents during the previous year. The Decatur price in October 1976 was 11¾ cents/lb., dry basis. This is 8⅓ cents/lb. in the sirup form. At this level, transportation costs become significant, perhaps explaining why some HFCS firms have temporarily pulled out of California. They were finding it difficult to maintain a 10-15% price differential under sugar prices. When produc-

tion costs are calculated, it appears that HFCS prices can always be less than United States sugar prices. Even to replace all imported refined cane sugar would require only 6% of the corn crop. We see that HFCS will always be with us, always be relatively cheap and plentiful.

First, I will review our work on the HFCS problem, then describe other efforts at the laboratory. Because of the extremely variable composition of honey and the highly refined nature of HFCS, the approach of choice to detect HFCS is to find constituents in it which are not found in honey at all, and then test suspected samples for the constituent or property. To use honey components, with all their variability, requires analysis for many of them with a statistical examination of probabilities of any particular combination occurring by chance. It is possible, but certainly not a preferred approach.

Last year I said that we had begun preliminary work on one approach, isotope ratio analysis, and had to drop it because the mass spectrometric instrumentation at our laboratory was not suitable for this analysis. This autumn, after several promising possibilities faded away as we stared at them, we decided to have another go at isotope ratio analysis. Since our equipment is unsuitable, we persuaded our Director to find the funds to go outside for the work. This he did, and we thank him for it. It has led to an absolute method to detect high fructose corn sirup when added to honey which cannot be circumvented. It is based on a fundamental difference in the atoms of carbon which make up the sugars of HFCS and those making up the components of all honeys we have examined. This phenomenon has come to light only recently as a result of basic research in plant physiology and biochemistry. It has been shown that there are two general groups of plants with respect to the ratio of their carbon isotopes of atomic weight 13 and 12. One

group, the more enriched in carbon 13, includes most of the grasses, lower plants, marine plants, and monocotyledons. The other group includes most flowering plants. Of course, it isn't that simple; there is a third group intermediate between them, and the known limits of each group overlap somewhat.

The mechanisms responsible for this difference are now fairly well understood: the three groups have differing photosynthetic mechanisms. The first group, including corn, uses the Hatch-Slack carbon cycle, while the other major group, including most flowering plants, uses the more familiar Calvin cycle. This means that each group uses different enzymes to fix carbon from atmospheric CO<sub>2</sub> into the plant constituents.

As I said earlier, the values of  $\delta^{13}\text{C}$  in the literature do fall into groups, but with a wide range for each group. For this approach to be useful to us in our problem, we needed to know what kind of averages and ranges in the ratio of carbon 13 to carbon 12 are found in honey and in corn sirup.

No information existed about this, so we selected, with the advice of our statistician, nearly 90 honey samples from the collection, to be as representative as possible of commercial United States honey. The cost of the analyses restricted us to using only a fraction of our samples. Several HFCS were included, of course, as well as a dozen mixtures. The analyses were done by a commercial laboratory in Cambridge, Mass. which specializes in stable isotope ratio analysis. The results were better than we had dared to expect: all the honeys fell into a rather narrow range centered on a value of about -25 per thousand. The corn sirup, as expected, averaged close to -10 per thousand. The variability was less for the honeys than for any honey constituent recorded in the literature. Mixtures fell between the pure materials.

What does this mean? It means that we now can test any sample and know

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if it contains any significant amount of HFCS or material of similar properties. Further, we believe, and hope to establish experimentally, that even if HFCS is fed to the bees and stored with honey we can detect it after extraction.

I hope that the beekeepers here today were listening to that last sentence. I'll say it again — We believe, and hope to establish experimentally, that even if HFCS is fed to the bees and stored with honey we can detect it after extraction. So if anyone uses HFCS for bee feed they had better be certain that appreciable amounts of stores from feeding are not mixed in with any surplus which is to be marketed. The test cannot tell just when the HFCS was mixed with honey — whether it was before or after extraction.

And while I'm on the subject it is only fair for me to say that one of the forms of sucrose or table sugar that is commonly used for bee feed, also responds to the test. So again, precautions must be taken that stored sugar from bee feeding is not mixed in with the surplus honey in any appreciable amount. Good beekeeping practice has never allowed this anyhow. The difference now is that it can cause a sample to be declared adulterated. In the eyes of the enforcement agencies, so-called honey made from sugar-feeding of bees is just as much adulterated as if the sugar were added later.

This test is somewhat expensive and there are only a very few laboratories that can carry it out. This means that

there is still a need for what I call a "screening test" — one that can be used in ordinary laboratories to select samples sufficiently suspicious to justify the isotope ratio test. This is what we have been doing and are continuing with.

Our work with the traces of complex carbohydrates in HFCS will lead, we hope, to suitable screening tests. It is still too soon to say. One problem is that in a sense we are shooting at a moving target; the corn sweetener industry is producing better (from their viewpoint) sirups all the time, making our job more difficult. This does not refer to the isotope ratio test, but only to prospective screening tests.

Another approach we are studying is the use of immunological testing. We are working with the ARS Pharmacology Laboratory in Berkeley to develop specific tests for the higher carbohydrates of HFCS. This is a very slow process which requires the development of an antiserum to a preparation from HFCS which is then supposed to indicate the presence of the HFCS substance in a mixture when tested with it. Although we have been on this for the best part of a year we do not presently know if this will be a useful approach. Now about other work in our laboratory.

The Food and Drug Administration has extended the Interagency Agreement with us until September 30. This funds a chemist who is analyzing the authentic honey samples, which you Federation members so generously provided, for sucrose and for HMF.

I also consult, on request, with FDA and State chemists and officials on problems with honey, as well as with Customs offices. The activity in this area appears to be considerably lessened from a year ago, which I hope is a good sign.

Now, another aspect of our work. As you probably know, the Honey Industry Council has provided funding for an analytical chemist stationed in our laboratory to strengthen our efforts by carrying out important work that otherwise we would not be able to do with our limited funds. Dr. Orest Rudyj has been with us for five months and has analyzed all of our samples for the amino acid proline, a constituent of all honey. Next, he will analyze for true protein. We greatly need data of this kind so that we can have additional information on the values and variability in honey which can be combined with other data already available to lead to ways to select suspicious samples for other testing. Dr. Rudyj is a careful, diligent worker and we are pleased to have him with us.

To summarize, this is where we are: Our earlier hopes that carbon isotope ratio analysis would provide the definitive answer to the HFCS problem have been realized. The nature of the test pretty much requires that some sort of preliminary screening test be used to select samples for that test. Three procedures we have been working on may qualify for the screening test, and additional work is needed to check these out. We are getting on with it.